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MAR 82 J V MARZIK, R KERSHAW, K DWIGHT

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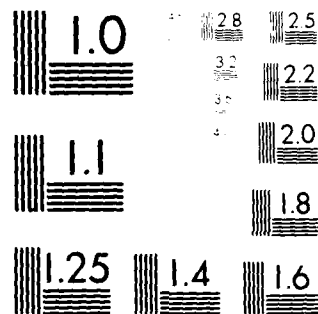
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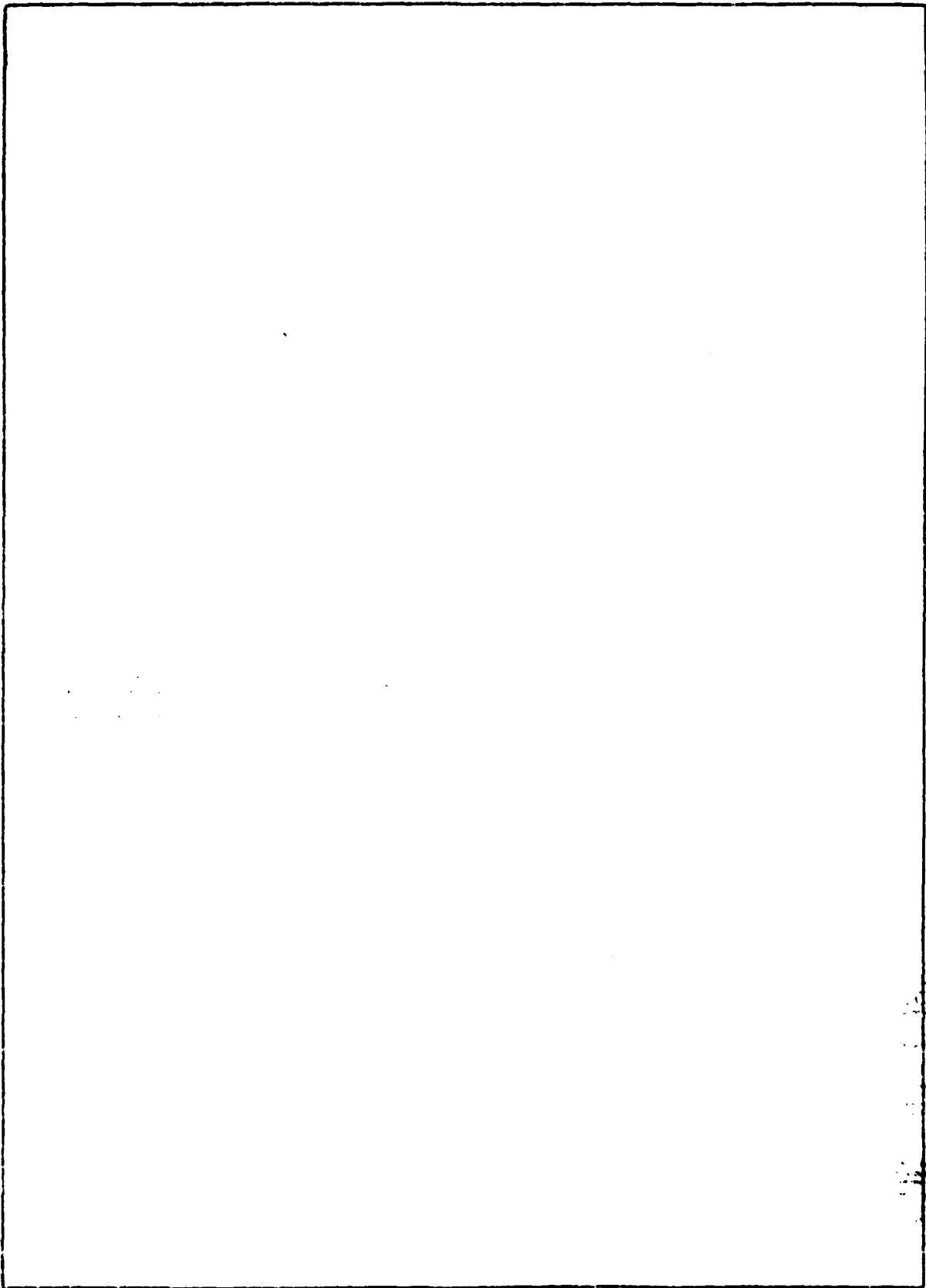
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TECHNICAL REPORT NO. 19

Preparation and Properties of PdPSe Single Crystals

by

J.V. Marzik, R. Kershaw, K. Dwight, and A. Wold

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INTRODUCTION

In recent years several investigators have studied the application of semiconductor electrodes in the construction of photoelectrochemical, photovoltaic, and electrophotosynthetic cells. There are several excellent reviews of these studies (1-3). Tributsch et al. (4-5) reported the use of MoS_2 and related semiconducting layer-type dichalcogenides in photoelectrochemical cells which show reasonably good efficiencies and long-term stability in aqueous solution, and since then, there have been several investigations of the photoelectrochemical behavior of layer-type transition metal compounds (e.g. 6-8).

Since PdPSe is a layer-type compound, it was of interest to undertake the synthesis of large single crystals and investigate their magnetic, electrical, optical, and (particularly) photoelectronic properties, since no semiconducting compound has been reported in which palladium acts as a photoactive center.

The synthesis of PdPSe was first reported by Bither et al. (9), and the structure determination of the isostructural PdPS was carried out by Jeitschko (10). PdPSe crystallizes with orthorhombic symmetry, space group Pbcn. It has a layer-type structure which is shown in Fig. 1. It can be seen that Pd is in square-planar coordination with two Se and two P atoms. (There are also weak interactions with Se and another Pd atom above and below the coordination plane, so the coordination geometry can be said to be that of an extremely elongated octahedron.) Phosphorus is tetrahedrally coordinated to two Pd, one Se, and one P atom, and selenium may be said to be tetrahedrally coordinated to two Pd and one P, with the fourth "bond" of the

tetrahedron being a weak interaction with a Pd atom from another layer. Each layer of PdPSe consists of a net of puckered pentagons which contain either two Pd, two P, and one Se atom or two Pd, two Se, and one P atom. Every other layer is connected by strong P-P bonds which form double layers of pentagon nets. These double layers are connected by weak Pd-Se interactions which are presumably broken upon cleavage of the crystal. Thus, the surface layer of PdPSe contains both Pd and Se atoms. This is different from most of the layer-type compounds which have the classical CdI_2 or MoS_2 -type structures consisting of layers of anion-metal-anion sandwiches that are loosely bound to one another by Van der Waals forces. Upon cleavage, the surface layer of these crystals solely contains the anion. The presence of palladium (a well-known catalyst for a large number of chemical reactions) on the surface of a photoactive crystal opens up the possibility of carrying out photocatalytic reactions on that surface.

EXPERIMENTAL

Synthesis

Single crystals of PdPSe were prepared from the elements by vapor sublimation. Palladium (Engelhard 99.99% pure) was pretreated by reduction at 900°C under 85%/15% Ar/H_2 gas for 16 hr. followed by heating for 4 hr. at 350°C under dynamic vacuum in order to remove hydrogen. Phosphorus (Leico 99.999% pure) and selenium (Gallard-Schlesinger 99.999% pure) were used as received. Stoichiometric amounts of the elements were sealed in a silica tube of dimensions 14mm OD x 12mm ID x 28cm in length which were evacuated to a pressure of $<5\mu\text{m Hg}$.

The tube was placed in a three-zone furnace (the third zone allowing good temperature control in the crystal growth zone), and the charge was prereacted for 24 hr. with the charge zone at 600°C, and the growth zone at 1000°C preventing transport of product. The furnace was then equilibrated at 880°C and programmed over 24-48 hours to give a temperature of 850°C in the charge zone and 800°C in the growth zone. Crystal growth took place in 5-7 days. Single crystals of PdPSe were silver-colored, thin rectangular platelets of sizes ranging from 1 to 50 mm² and thicknesses ranging from 5-50 μm. The crystals had a graphite-like habit.

X-ray Analysis

X-ray diffraction patterns of small single crystals were obtained using a Gandolfi camera (Blake Industries, Model D1100) and CuKα radiation. Lattice parameters were obtained by least-squares refinement of the diffraction data.

Magnetic Measurements

Magnetic susceptibility was measured using a Faraday balance (11) over the range from liquid nitrogen to room temperature at a field strength of 10.4 kOe. Honda-Owen (field dependency) plots were also made to determine the presence or absence of ferromagnetic impurities. The data were then corrected for core diamagnetism (12).

Electrical Measurements

Resistivity and DC Hall effect measurements were made on single crystals of PdPSe using the van der Pauw technique (13). Contacts were made by the

ultrasonic soldering of indium directly onto the samples, and their ohmic behavior was established by measuring their current-voltage characteristics. Activation energy of resistivity E_a (defined for semiconductors by $\rho = \rho_0 \exp(E_a/kT)$ where ρ = resistivity, ρ_0 is a constant, T = temperature (K), and k = Boltzmann constant) was determined by measuring ρ as a function of T .

Optical Measurements

Optical absorption measurements were carried out on thin (5-25 μ m) single crystals with a Cary 17 spectrophotometer. The absorption coefficient α was calculated from the transmission ratio T using the expression $T = (1-R)^2 e^{-\alpha t} [1-R^2 e^{-2\alpha t}]^{-1}$ where t is the sample thickness and R is the reflectivity calculated from the above expression by assuming $\alpha \approx 0$ in the part of the spectrum far removed from the absorption edge.

Electrode Preparation

Photoelectrodes were prepared by evaporating a thin film of gold on the back of the single crystals of PdPSe in order to provide good electrical contact. For mechanical support, the crystal was affixed to a disk of platinum foil with a drop of silver paint. The platinum foil was soldered to the platinum electrode wire which was sealed inside a pyrex tube. An insulating resin (Microstop, Michigan Chrome Chemical Corp.) was applied to the platinum foil and wire so that only the front surface of the crystal was in electrical contact with the electrolyte solution.

Photoelectronic Measurements

Photoelectronic measurements were carried out with a 150-W xenon lamp, a monochromator (Oriel Model 7240), a glass cell with a quartz window, and a current amplifier as previously described (14). A tungsten iodide lamp was used for wavelength dependency measurements. The quantum efficiency η was determined by dividing the current through the cell by the incident photon flux, measured with a calibrated Si photodiode. The electrolyte used was an aqueous solution of 0.05M NaI/0.002M I_2 /0.05M H_2SO_4 . This most likely results in the I^-/I_3^- couple as the electroactive species in solution (6). The electrolyte was purged of dissolved oxygen by continuous bubbling of nitrogen gas. At 0.0V bias with respect to the counter electrode, the potential of the working electrode was 0.335V versus SCE.

RESULTS AND DISCUSSION

X-ray analysis shows that PdPSe has orthorhombic diffraction symmetry. Cell dimensions $a = 13.59(3)\text{\AA}$, $b = 5.81(1)\text{\AA}$, and $c = 5.85(1)\text{\AA}$, obtained by least-squares refinement of Gandolfi x-ray data, agree well with those obtained by Bither et al. (9).

The electrical properties of PdPSe are summarized in Fig. 2 and Table I. This material is a semiconductor with a room temperature resistivity of 70 ohm-cm and an activation energy of resistivity of 0.32 eV. DC Hall and Seebeck measurements show that the sign of the majority carrier is negative, which is indicative of an n-type semiconductor. Hall measurements indicate a relatively low concentration of charge carriers ($2.4 \times 10^{15} \text{ cm}^{-3}$) with moderately high mobility ($34 \text{ cm}^2 \text{ V}^{-1} \text{ sec}^{-1}$).

The magnetic data for PdPSe (see Fig. 3) indicate a very weak temperature-independent paramagnetism (Pauli paramagnetic behavior). The magnetic behavior is indicative of the presence of some delocalization of the electrons. This is consistent with its comparatively low resistivity value. Measurements of the magnetic behavior of the isostructural compound PdPS show it to be diamagnetic (9) and, as expected from a material in which all the electrons are spin-paired, the measured value of the resistivity at room temperature is very high ($9 \times 10^7 \text{ ohm-cm}$).

The photoresponse observed for PdPSe is represented in Fig. 4 where the photocurrents obtained in "white" light are plotted against the anode potential measured with respect to a saturated calomel electrode (SCE). The

onset of the photocurrent indicates a flat-band potential of approximately 0.1 V vs SCE. Anodic dark current was negligible ($0-0.05\text{mA/cm}^2$) compared to the observed anodic photocurrent. When the PdPSe electrode was biased cathodically with respect to the counter electrode, a somewhat higher cathodic dark current ($0-1\text{ mA/cm}^2$) was observed due to the diode character of the semiconductor-electrolyte junction; even so, a significantly larger anodic photocurrent was observed down to the onset of photocurrent at 0.1 V vs SCE. The photocurrent is due to the reversible oxidation of I^- to I_3^- (or I_2) since a cell containing only $0.05\text{M H}_2\text{SO}_4$ as the electrolyte showed negligible photocurrent.

The quantum efficiency η (in electrons/photon of PdPSe as measured at an anode potential of 0.335 V vs SCE) is plotted in Fig. 5. Experiments measuring the absorption due to the electrolyte showed it to be negligible in the spectral region pictured. The onset of photocurrent corresponds very closely to the absorption edge of 960 nm which was measured from the optical absorption spectrum of PdPSe. There is a steady increase in η until it levels off at a value of about 60% at 600 nm. This value does not take into account the amount of light lost by reflectance off the surface of PdPSe. The reflectivity of PdPSe was calculated to be 0.37 from the absorption spectrum. Correcting for the reflectivity value results in quantum efficiencies of 90-100% below 600 nm. Below 530 nm, the quantum efficiency decreases somewhat, a phenomenon which has been observed for other layer-type materials (4-7,15). It has been suggested (5,15) that the photoeffect observed in layer-type transition metal dichalcogenides is due to d-d electron transitions. Such a model would be

consistent with the observed decrease in photocurrent at the shorter wavelengths. The electron delocalization is evidenced by the Pauli paramagnetism of PdPSe and the moderately high mobility given in Table I. Furthermore, the stability of the PdPSe photoelectrodes, which were used reproducibly for several experiments, also suggests that the photoeffect is due to d-d transitions rather than the breaking of metal-anion bonds (16).

Analysis of the spectral response data can yield values for various energy transitions (17). Accordingly, the quantity $(nh\nu)^{.5}$ is plotted as a function of the photon energy in Fig. 6. This analysis yields a lowest-energy indirect optical band gap of 1.28(1) eV. This is in good agreement with the band gap obtained by analyzing the optical absorption spectrum. Fig. 7 is a plot of $(\alpha h\nu)^{.5}$ as a function of photon energy which results in a lowest-energy indirect optical band gap of 1.29(1) eV. In addition, from the data presented in Fig. 6, a higher energy band transition can be estimated, as indicated by a sudden increase in slope. This transition occurs at 1.48(5) eV.

SUMMARY AND CONCLUSIONS

Large single crystals of PdPSe have been grown by vapor sublimation and several of their properties investigated. PdPSe was found to be an n-type semiconductor with weak Pauli paramagnetic behavior. DC Hall measurements indicate a low concentration of highly mobile charge carriers. Photoelectronic measurements show PdPSe to be a photoactive material with high quantum efficiencies below 800 nm. Optical and photoelectronic measurements agree closely, giving a lowest-energy indirect optical band gap of 1.28(2) eV. Analysis of

the photoelectronic spectral response also results in a measured higher-energy indirect transition at 1.48(5) eV.

Because of its unique layer structure, the surface of the natural cleavage plane of a PdPSe crystal contains both Pd and Se atoms. Since palladium is present at the cleavage plane of this photoactive material, there is the possibility that this or similar palladium-containing photoactive materials may provide surfaces on which photocatalytic reactions can take place.

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TABLE I

Electrical Properties of PdPSe

	<u>ρ (ohm-cm)^a</u>	<u>E_a (eV)</u>	<u>n (cm⁻³)</u>	<u>μ_H (cm²V⁻¹sec⁻¹)</u>
This work	70	0.32 ^b	2.4×10^{15}	34
Literature ^d	30	0.15 ^c	-----	---

a 300 K

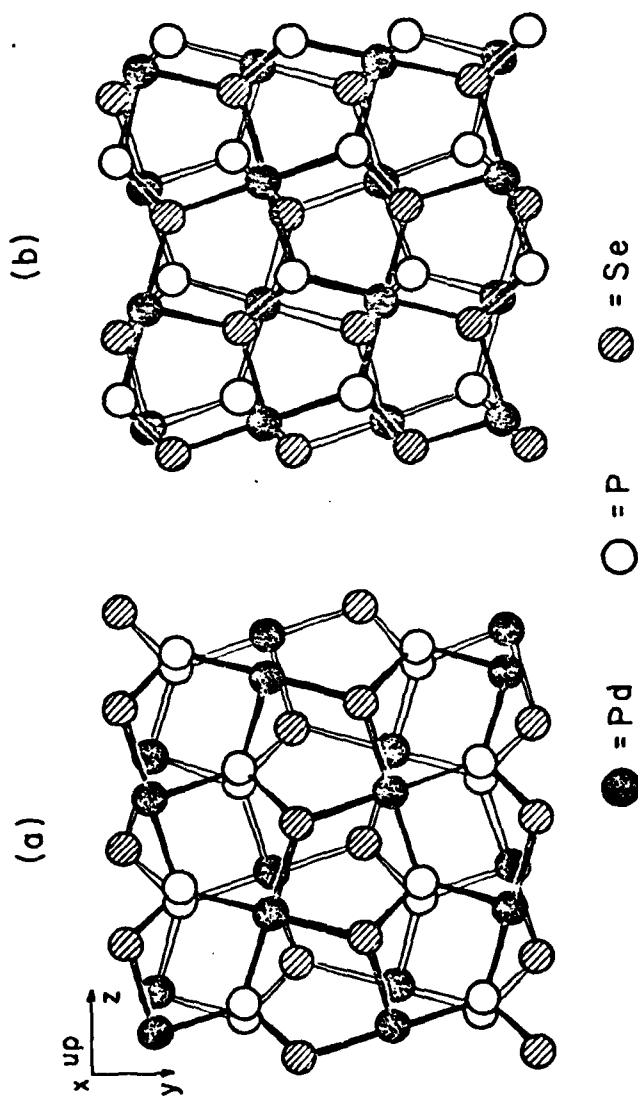
b 200-320 K

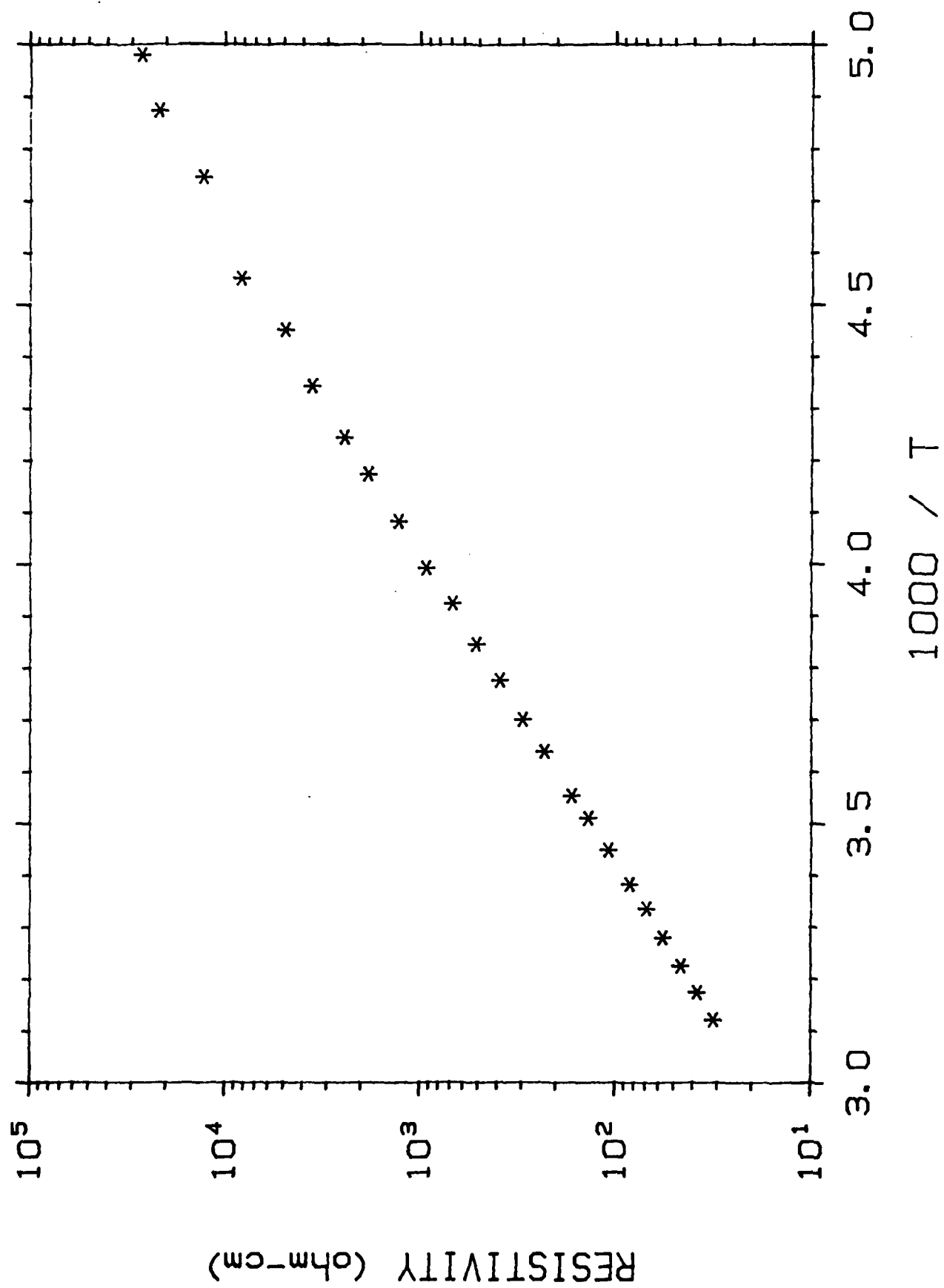
c 300-400 K

d Reference 9

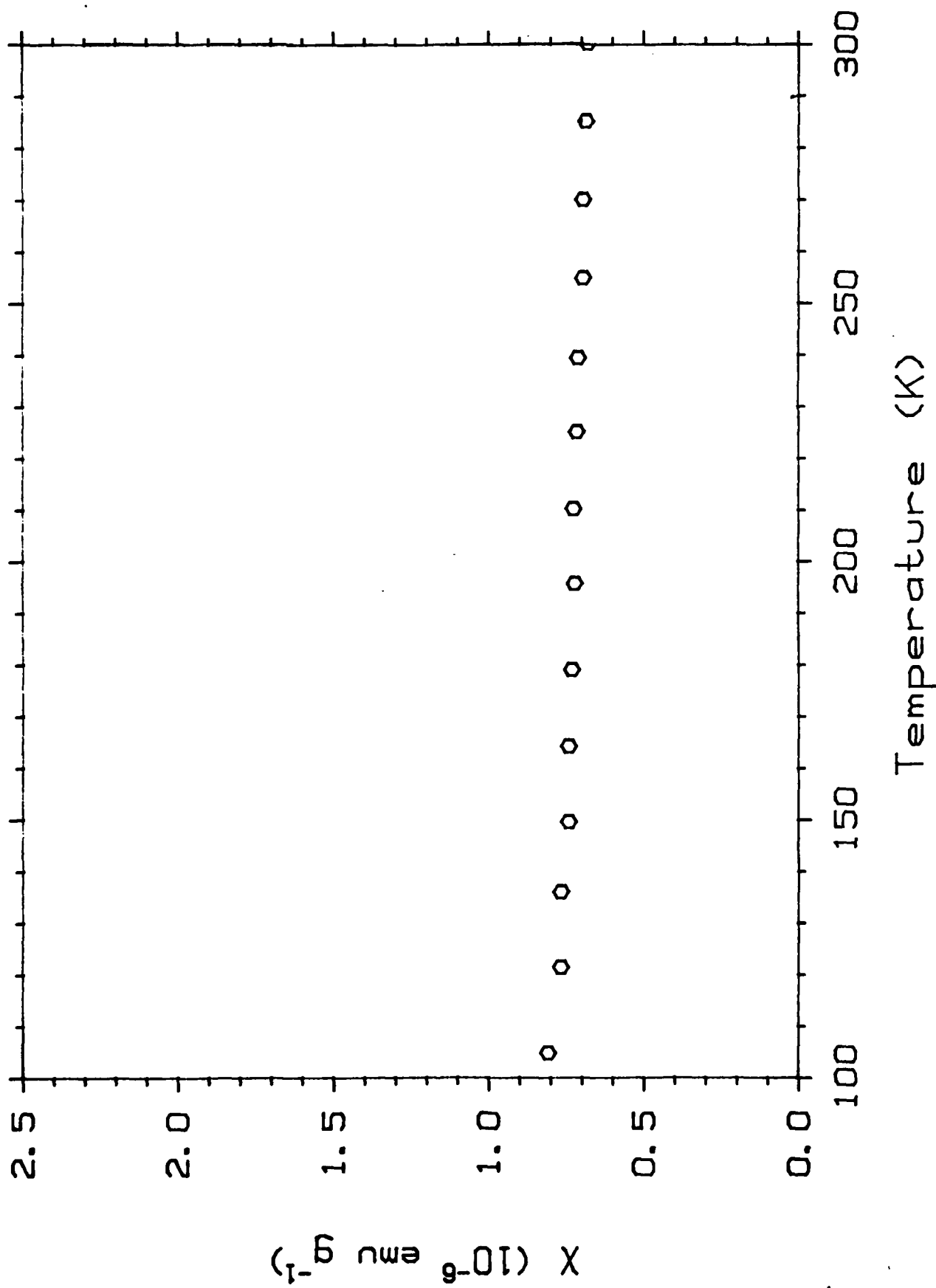
FIGURE CAPTIONS

- Fig. 1. Relative positions of atomic layers in the PdPSe structure. Atoms in the upper layers are connected by solid lines and those in the lower layers by double lines. The double layers of PdPSe shown in (a) are connected by P-P bonds and weaker Pd-Pd interactions. (b) illustrates the relative positions of two layers (the "top" of one double layer and the "bottom" of another) which are connected by weak Pd-Se interactions.
- Fig. 2. Resistivity of PdPSe plotted as a function of temperature.
- Fig. 3. Magnetic susceptibility of PdPSe plotted as a function of temperature.
- Fig. 4. Variation of photocurrent with anode potential under "white" xenon arc irradiation of 1.0 W/cm^2 in $0.05\text{M NaI} / 0.002\text{M I}_2 / 0.05\text{M H}_2\text{SO}_4$ electrolyte solution.
- Fig. 5. Spectral variation of the quantum efficiency obtained at an anode potential of 0.335 V vs SCE in $0.05 \text{ NaI} / 0.002\text{M I}_2 / 0.05\text{M H}_2\text{SO}_4$.
- Fig. 6. Indirect band gap analysis for PdPSe showing transitions at $1.28(1)$ and $1.48(5) \text{ eV}$.
- Fig. 7. Analysis of the optical absorption spectrum of PdPSe in the region of the absorption edge, giving a lowest-energy indirect optical band gap of $1.29(1) \text{ eV}$.

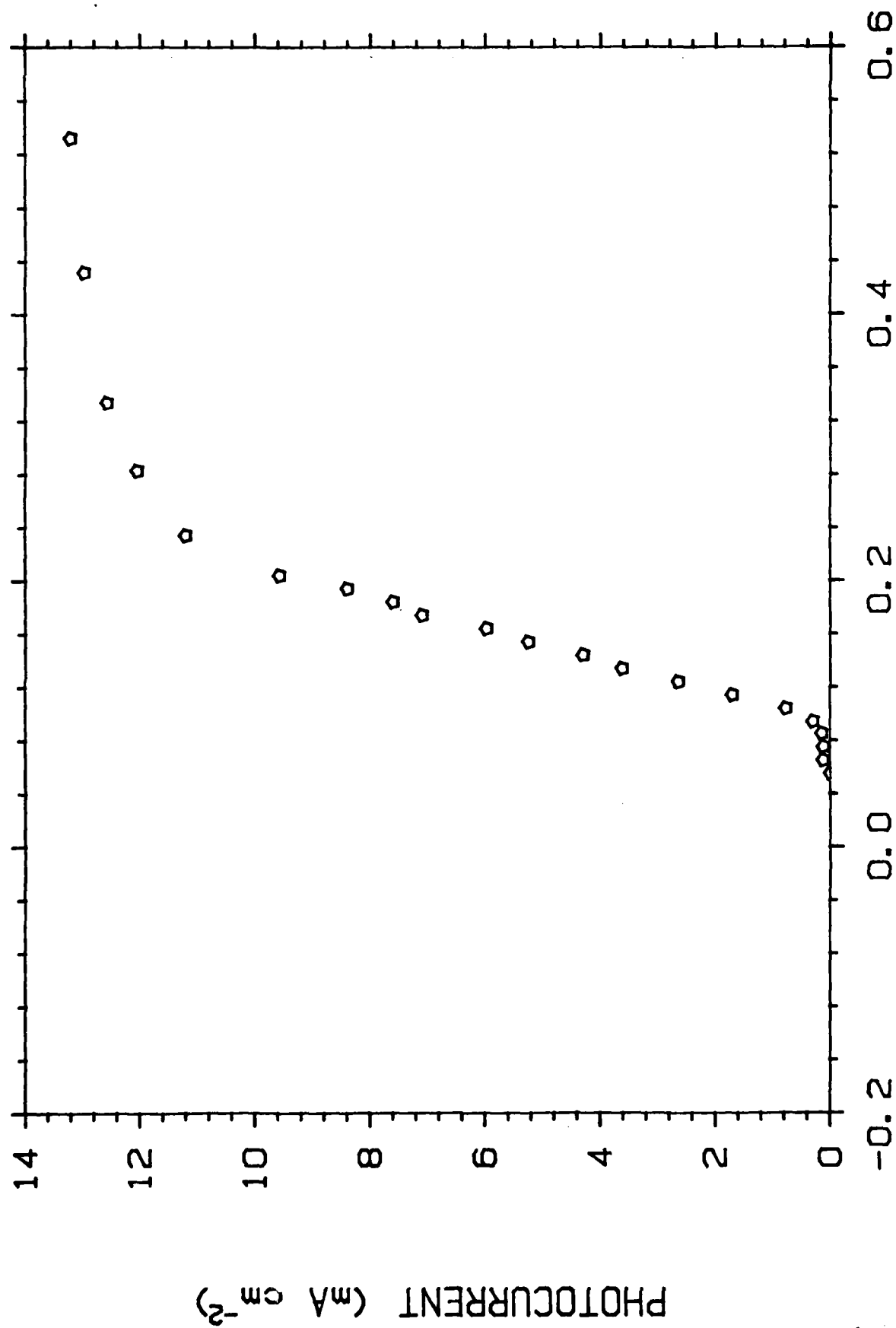




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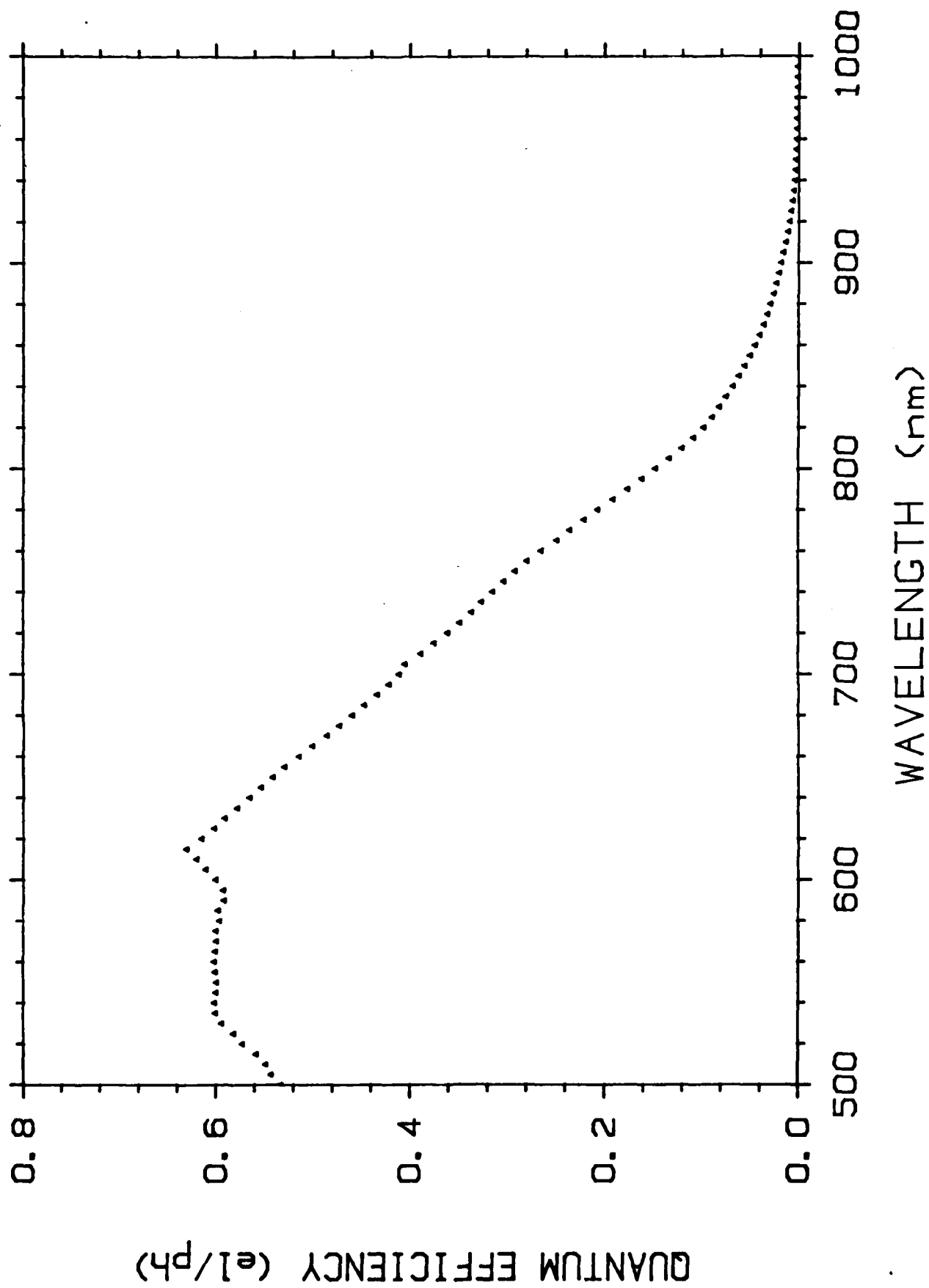


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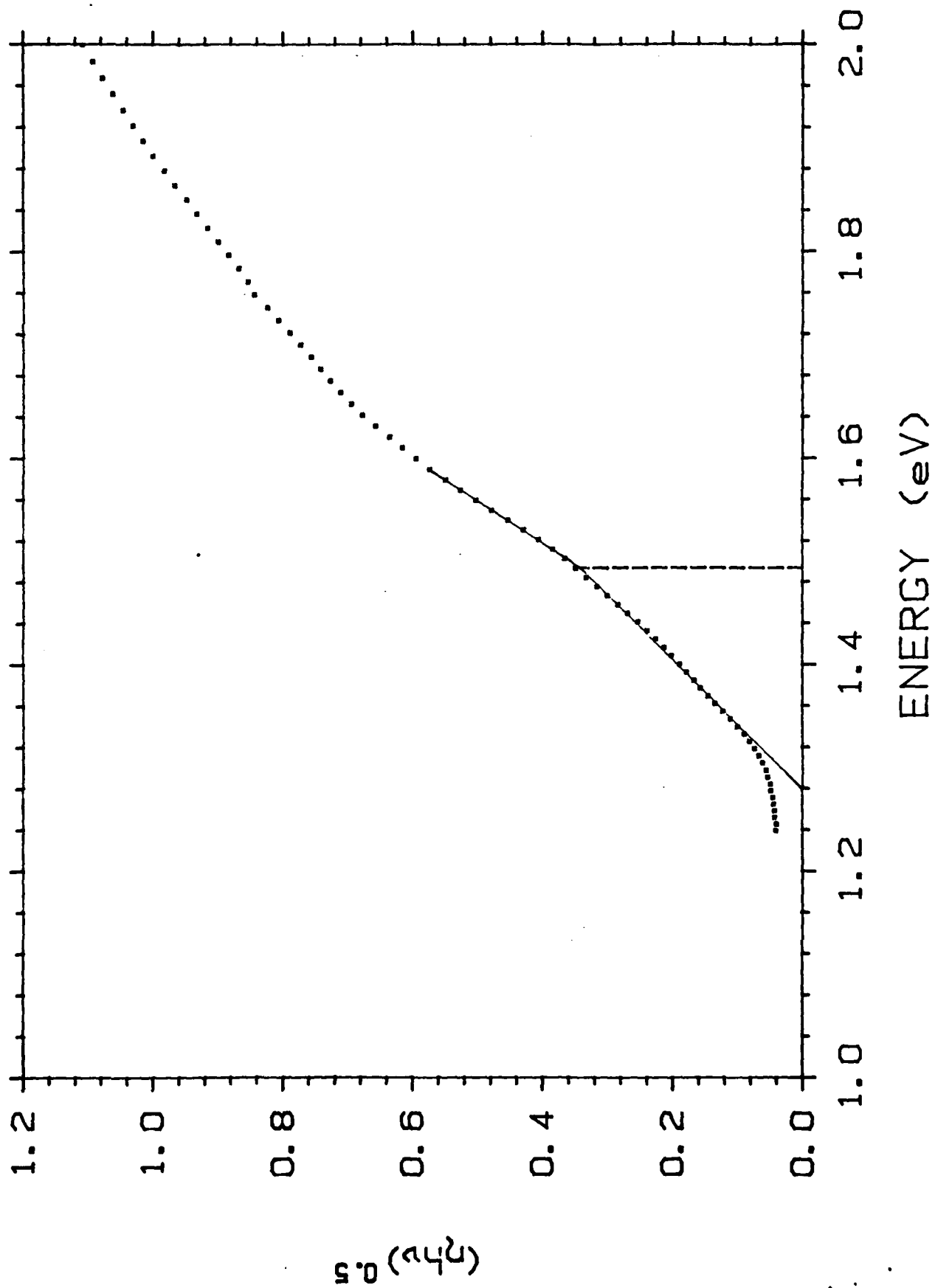


ANODE POTENTIAL vs SCE (volts)

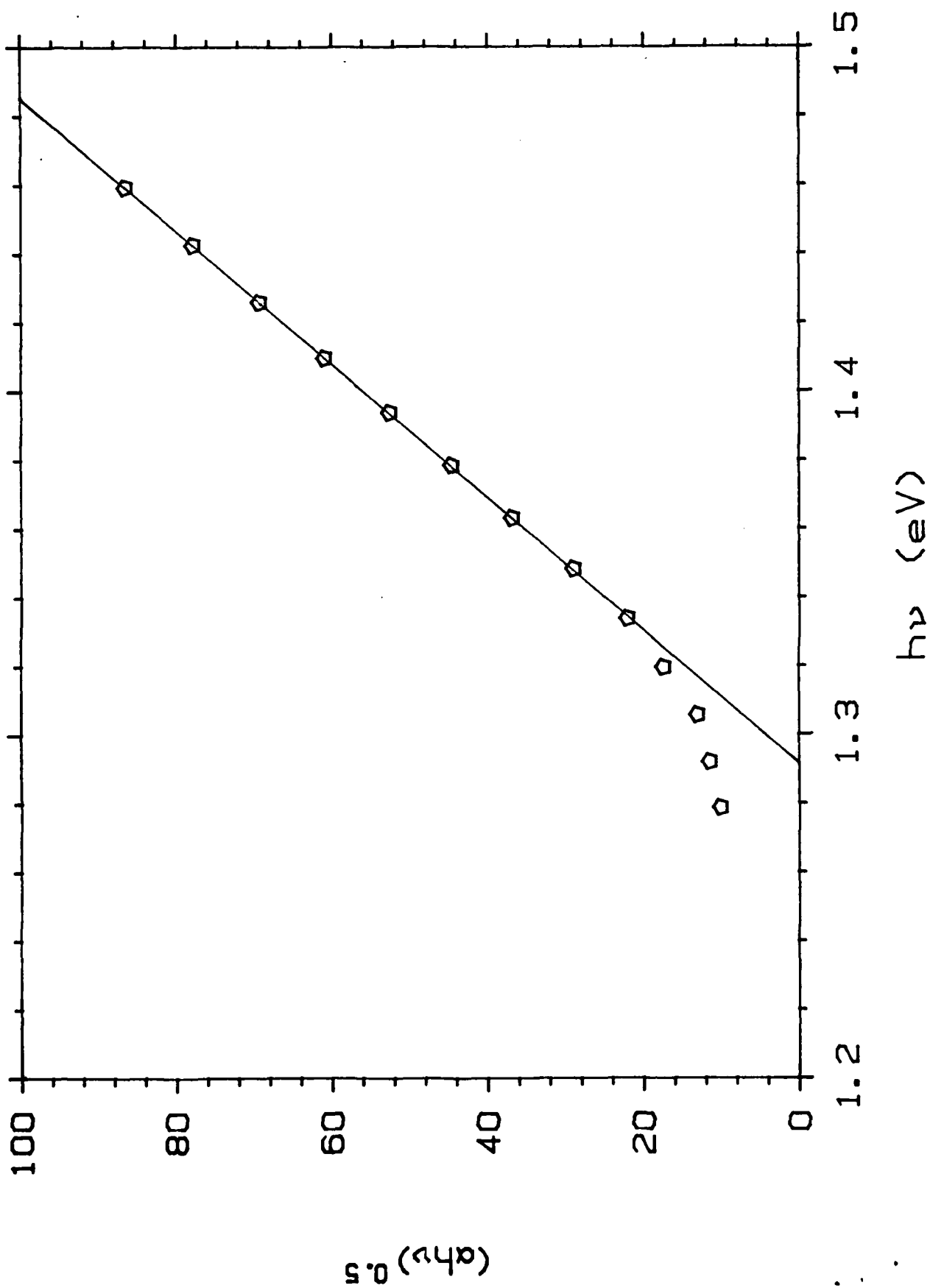
SPECTRAL RESPONSE



PdPSe



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